MIKOVINY SÁMUEL DOCTORAL SCHOOL OF EARTH SCIENCE

THESES OF DOCTORAL DISSERTATION

INVASTIGATION OF LOW PERMEABILITY HYDROCARBON RESERVOIR PROPERTIES

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I. SCIENTIFIC BACKGROUND AND AIMS

In the last two decades, especially in the last ten years, significant attention has been paid to projects and industrial investments exploring the possibilities of exploiting unconventional hydrocarbon reservoirs. As part of sustainable development and security of energy supply, many well-known domestic and international universities and research institutes investigating methods and operating mechanisms that can describe the petrophysical and mechanical characteristics of such individual systems.

Shale gas reserves are one of the most abundant sources of natural gas in the world, largely untapped and found on most continents. Developments aimed at exploiting resources began in North America, predominantly in the United States, leading to a boom in gas and oil production. Despite significant advances in production from these reservoirs, their behavior and characteristics are still less well known. The applicability of hydraulic fracturing operations, the description of the flow mechanism in the rock matrix and cracks, and short - to - long - term productivity are still open questions.

Permeability is one of the most important parameters controlling production. This property of rock is a measure of fluid conductivity. The parameter is determined by laboratory tests, geophysical logs, correlations in the case of conventional reservoirs. However, in rocks with exceptionally low permeability, measuring this parameter is extremely challenging and does not follow the physical laws already used in conventional rocks. Based on *Klinkenberg's (1941)* modified Darcy equation, the so-called absolute permeability of a porous medium can be estimated from a series of gas permeability measurements. However, the pressure dependence of permeability is much more complex in closed systems, so the Klinkenberg correlation, as mentioned above, gives erroneous results *(Ziarani and Aguilera, 2012; Moghadam and Chalatyrnyk, 2015)*.

Several significant studies on the permeability of shales have been published, both theoretically and analytically *(Moghadam and Chalaturnyk, 2014; Sakhaee-Pour and Bryant, 2012; Javadpour et al., 2007; Ziarani and Aguilera, 2012; Roy et al., 2003)*. *Moghadam and Chalaturnyk (2014)* proposed changing the zero – velocity gradient as an extension of the original Klinkenberg equation if the flow falls in the ‘slip’ range. The proposed equation is expected to be able to estimate gas permeability in ‘slip’ and ‘early transient’ flow systems for shale. *Ziarani and Aguilera (2012)* investigated a second-order Knudsen correlation to calculate gas permeability and concluded that the Klinkenberg correlation underestimates the increase in permeability due to slip flow. *Rahmanian et al. (2013)* hypothesized that the flow in shales consists of a viscous and free molecular flow component. *Singh et al. (2014)* also proposed an equation to determine apparent permeability based on Darcy and Knudsen flow. These equations for determining apparent permeability are based on a number of (typically empirical) parameters that require laboratory measurements to determine. Their application is therefore difficult, as some parameters may not be measurable directly in the laboratory. In addition, to compare the apparent permeability to the Knudsen number, pore diameter values are required, which are often not available, and vary depending on the test conditions, such as the average effective voltage. No consistency can be detected between the methods and there are occasional differences in the measured permeability values. *(Gasparik M. et al., 2015; Rushing et al., 2004; Sinha et al., 2012; Clarkson et al., 2012)*.

It is clear from the above that the field has encountered extremely complex problems, the significance of which is by no means questionable from a scientific point of view. The scientific program I designed, aimed the expansion and refinement of the measurement technology available at the Research Institute of Applied Earth Sciences by mathematical model application based on the examination then the results of the investigation of Hungarian marl and compacted or tight sandstone deposits with low permeability and poor recovery factors. The result will greatly contribute to facilitating the work of the Research Institute and other organizations engaged in laboratory activities in unconventional research areas.

A review of the relevant literature, as well as industrial documentation and production data, made it necessary to carry out a specific testing program, considering the Hungarian industrial experience. In the practical adaptation of new hypotheses and models to be developed, special attention should be paid to their validation using existing, available tools. Utilizing the advantageous properties of the above methods, I carried out the investigations, the interpretation of the measurements, the method developments and obtained new scientific results.

II. ACCOMPLISHED INVESTIGATIONS AND INTERPRETATIONS

The toolset of the Research Institute of Applied Earth Sciences has several instruments that are suitable for determining the petrophysical parameters of rocks. The instrumentation of the Research Institute is able to measure both basic and special (e.g. relative permeability) parameters over a wide range. It is important to note that my research presented in this dissertation has been a major challenge in many cases. The Quantachrome Ultrapyc-1200e helium porosimeter and the Pascal 140 and 440 Mercury Injection Instruments (HPMI - High

Pressure Mercury Injection) were used for porosity measurements. In the case of helium porosity measurement, the porosity value can be calculated indirectly from the measured solid volume and / or density value, which requires a preliminary mass measurement of the samples. The equations integrated in the program of the HPMI instrument basically derive the results from the measured volumes, thus, in this case, it is not necessary to perform separate calculations, the instrument gives the results directly. A number of other parameters were determined using mercury injection measurement, such as specific surface area, apparent density, cumulative volume, and pore-throat radii and distribution, which formed a significant part of the analyzes.

For a wide and accurate determination of permeability, the Research Institute has developed a so-called N_2 gas permeameter in accordance with the API RP 40 *(American Petroleum Institute, 1998)* standard, a Pressure Pulse Decay (PPD) instrument based also on the mentioned standard, and Ultra-Perm 610™ and Nano-Perm™ gas permeameters, developed and marketed by Core Laboratories™, were used in the measurement program.

My research has focused primarily on tight sandstone and marl rocks. The selection of the sample groups and the compilation of the measurement program were fundamentally based on the rock samples accessibility and availability at the Research Institute.

At the beginning of the measurement program, the porosity of 194 unconventional rock samples measured with helium and the so-called 'pre-measured' permeability values determined with nitrogen by a conventional N_2 permeameter were measured for 176 samples. In line with my expectations, supported by preliminary measurements, the available conventional tools were suitable for determining porosity, but the range of tools available at the beginning of the research program to determine permeability with scientific accuracy was especially limited.

During the compilation of the measurement program, I tried to maximize the acquisition of information as much as possible, and in parallel to optimize the number of sample destructions caused by damaging / destructive measurements. 47 of the 62 tight sandstone samples were suitable for PPD measurements.

From the processing of the HPMI measurement results, I made several very clear and quick-to-review interpretations. For each sample examined by HPMI measurements, standard pore-throat distribution curves, pore-throat radius distribution curves as a function of the percentage of mercury injection and capillary pressure curves were plotted. Threshold pressure values were determined semi-graphically.

After the production of the basic data, I examined their correlations in order to make generalizable statements about the petrophysical properties of the studied formations (Szolnok and Endrődi). The relationship between porosity and permeability is known to be a long and continuously researched field in petrophysics. Starting from the fact that the order of magnitude of the permeability, in the case of tight sandstones, is mostly influenced by the presence and distribution of larger pores, I examined the pore space distributions. I came to the conclusion that it is not worth looking for a permeability relationship with the absolute value of porosity, but with some characteristic fraction of it. In the search for correlations, I discovered a very strong relationship between the volume formed by pores with a radius greater than 0.2 micrometers and permeability.

Adapting the theoretical approach of *Javadpour et al. (2007, 2009)* based on my simulation results, it can be said that the simulations performed with synthetic samples built from capillary bundles show an outstanding match with the measurement results even without the correction of the radius distribution of the pore throats.

By adapting the water film ring theory of *Yu-Liang Su et al. (2020)*, I developed a model describing the applicability of a "purified" and measurement-validated synthetic model specimen of a capillary tube bundle to determine connate water saturation.

I have described a strong relationship that can be easily applied in practice between the so-called reduced porosity I have introduced, the threshold pressure and connate water saturations belonging to a critical radius of 0.2 micrometers in the case of tight sandstones originated from Szolnok Formation.

The capillary tube bundle model describes the behavior of a standard "plug" sample during a measurement very well which is supported by measurement data made by the Research Institute. I have made a correlation to determine the apparent and Darcy permeability of the sample, as well as the permeability of standard, plug-size samples with cracks and thus significantly different measured results can be also determined by careful application of the model.

In the case of marl and calcareous marl type rocks, I also prepared the synthetic specimens from the pore-throat distribution curves based on HPMI measurements. After performing the simulations, I observed a strange phenomenon. In contrast to the sandstone simulation experiments, where minimal corrections had to be applied if needed, the mass flows of the marl samples measured and calculated without fitting were extremely different. The measurement results and the interpretation of the simulations provided further evidence and confirmed my hypothesis that both small-sized and plug size marl samples acquire / suffer significant surface and even sample-wide (microcrack) damage partly during storage and partly during sample preparation.

Given that the flow rate and permeability values measured with the Nano-Perm™ device in the validated model can only be obtained with a strong correction during fitting, I concluded that this measured permeability is the resultant of the intact inner "core" and the permeability of a "crust" which gives a much better or higher permeability value due to surface damage.

III. NEW SCIENTIFIC RESULTS

Thesis 1

Determination of permeability of unconventional rock samples using the results of high-pressure mercury injection (HPMI) measurements

Using the correlations, I have proposed, the absolute permeability of the tight sandstones of the Szolnok Formation can be determined with practical accuracy with HPMI measurement data. (N.P. Szabó et.al., 2021)

During the examination of the poro-perm relationships of the rock samples with typically unfavorable petrophysical properties from the Szolnok Sandstone Formation, I concluded that a well-established functional relationship between the absolute value of porosity and the absolute permeability cannot be revealed for unconventional sandstones.

I revealed a connection using measurement results for the correlation between the most frequent pore throat radius (MFPR) and the absolute permeability, which is described by the following relation:

$$k_{abs} = 25{,}746\ MFPR + 0{,}4959 \qquad R^2 = 0{,}7313$$

I discovered a strong relationship between the radius of the pore tubes with 15% mercury saturation (r_{15}) calculated from the HPMI measurement and the logarithm based on tens of the absolute permeability, which is described by the following relation:

$$\log k_{abs} = 2{,}8361\ r_{15} - 0{,}206 \qquad R^2 = 0{,}848$$

I invented a very strong relationship between the pore volume (reduced porosity: $\phi_{R0,2}$) formed by pores with a radius of more than 0.2 micrometers and the absolute permeability described by the following equation:

$$k_{abs} = 1{,}3571 e^{0{,}3345 \phi_{R0,2}} \qquad R^2 = 0{,}8959$$

Thesis 2

Development of a synthetic model sample to study gas flow from petrophysical properties in a porous medium

With flow rate measurements performed on intact rock samples, I proved that the results of the simulations with the synthetic samples built from the capillary bundles I proposed show excellent match with the measurement results (F. Remeczki, 2020b).

Based on the information from routine (RCA - Routine Core Analysis) measurements of small size rock samples from unconventional hydrocarbon reservoirs, a synthetic standard (plug size) model sample consisting of pertophysical properties can be created using kinetic gas theory under the validity conditions I propose which is suitable to study of material transport (gas) processes in the rock.

The synthetic sample is a model of capillary bundles in which the pore throat distribution curve of the HPMI measurement gives the diameter and size distribution of the capillaries and the number of capillary tubes in the bundle for a given capillary diameter.

Model input data	Sample data: HPMI porosity, HPMI pore throat radius distribution, and relative volume percentage. Gas used for measurement: molecular diameter, molar mass, dynamic viscosity and density of the gas under measurement conditions. From the measurement of the effective permeability of a standard-sized sample meeting the similarity criteria: the average pressure, the pressure difference and the flow rate for the given pressure difference, the sample length and diameter.
Model results	To every capillary bundle: Type of flow system defined by Knudsen number and flow rate

Thesis 3

Determination of connate water saturation with the application of synthetic model sample

I found that the sensitivity of the connate water saturation to the radius value of the critical pore tubes, as well as the pressure and temperature dependence of the connate water saturation can be investigated with the model. To do so, I created a formula suitable for calculating the saturation of the connate water of the model sample (F. Remeczki, 2020d; F. Remeczki, 2021a)

A preferred pore throat radius (critical pore throat radius: r_c) for the ascending series of equivalent capillaries by diameter was taken as the maximum value which serves as boundary between completely saturated and unsaturated capillaries. By considering the unsaturated capillaries that have larger radius than the critical one which have the presence of connate water as a water film rings (δ) formed on their walls, using the model, the connate water saturation (S_{wi}) of the synthetic model sample can be calculated by the following relationship:

$$S_{wi} = 1 - \frac{\sum_{j=c}^{n} \left(r_j - \delta_{j-c+1}\right)^2 \pi\ L\ N_{Cj}}{\sum_{j=1}^{n} r_j{}^2\ \pi\ L\ N_{Cj}}$$

Thesis 4

Estimation of petrophysical properties using calculations results of a synthetic model sample based on HPMI data

Knowing the threshold pressure determined semi-graphically from the capillary pressure curves, the connate water saturation of the tight sandstones and marls can be calculated with the accuracy corresponding to practical aspects using the formulas I have created.

Utilizing the hybrid of traditional methods (RCA) and the new synthetic model, I introduced an approximate function between the reduced porosity values I defined ($\phi_{R0,2}$) and the connate water saturations ($S_{wi0,2}$) for a critical radius of 0.2 micrometers, which is described by the correlation below:

$$S_{wi0,2} = -5{,}7908\ \phi_{R0,2} + 94{,}453 \qquad R^2 = 0{,}9493$$

Appling the correlation, the connate water saturation of the samples of both the Szolnok sandstone and the Endrőd marl rocks can be determined with an accuracy corresponding to practical aspects, for which the availability of the data of HPMI measurements performed on small samples is sufficient.

I introduced a function between the threshold pressure (p_{th}) of the Szolnok tight sandstones and the connate water saturation ($S_{wi0.2}$) belonging to a critical radius of 0.2 micrometer, which is described by the following relation:

$$S_{wi0,2} = 0{,}0071\ p_{th}^2 + 0{,}8809\ p_{th} + 50{,}806 \qquad R^2 = 0{,}9076$$

Thesis 5

Determination of the permeability of the synthetic model sample

With the new synthetic sample model method I propose, both apparent (k_{app}) and Darcy (k_D) permeabilities can be determined in capillary tubes of different radii. Applying the calculation method to the capillary bundles of the model sample, the values I call the weighted permeability can be determined for each pore throat radius. I proved by measurement results that the following relations are valid for the apparent and Darcy permeabilities determination *with the parallel coupling of the permeabilities calculated for each capillary tube: (F. Remeczki et. al., 2020; F. Remeczki and G. Horváth, 2021)*

$$k_{appminta} = \frac{\sum_{j=1}^{n}\left(k_{app_j} r_j^2 \, Nc_j \, \pi\right)}{\sum_{j=1}^{n} r_j^2 \, Nc_j \, \pi}$$

$$k_{Dminta} = \frac{\sum_{j=1}^{n}\left(k_{D_j} r_j^2 \, Nc_j \, \pi\right)}{\sum_{j=1}^{n} r_j^2 \, Nc_j \, \pi}$$

Thesis 6

A novel approach to permeability of marls – Crust Model Theory

I created the Crust Model to support a novel approach to the permeability of marl rocks. The way this synthetic sample model was created made it suitable to study the phenomenon, the fitting necessary to characterize the damaged 'crust' with an equivalent capillary bundle, and the determination of the gas flow characteristics in the intact 'core' (flow type, flow rate and permeability) (F. Remeczki, 2020a, 2020c, 2021b).

I found that in the case of marl samples, the measurement results of the standard plug-sized samples and the simulations performed on the synthetic sample built from the pore-throat radius distribution of the HPMI measurements show orders of magnitude differences compared to each other in both flow rates and permeability values despite the same conditions.

I also found that in the case of the Endrőd marl samples, the radius distribution of the pore tubes from the HPMI measurement is not representative for the larger, standard-sized sample, and that the measured permeabilities do not characterize the pore space with given radius distribution.

I came to the conclusion that in the case of the Endrőd marl-type rocks the simulations performed on the synthetic model sample require the removal of the so-called damaged zone and the introduction of a certain equivalent capillary, thus creating the behavior of the original sample under measurement conditions.

Regarding the permeability of the Endrőd marl-type rocks, I proposed a new theoretical approach, according to which the permeability measured on a standard size rock sample cannot be considered as a representative value for the total volume of the sample and thus for the investigated rock type. The measured permeability of a standard-sized specimen shall be considered as the resultant of the permeability of the intact inner core and the permeability of the mantle with altered permeability due to surface damage.

V. PRACTICAL APPLICATION OF THE RESULTS

Using formation-specific poro-perm relationships, a faster and more cost-effective estimation of rock permeability is obtained without measurements.

By using the synthetic model sample presented in the dissertation, connate water saturation at different p, T, Δp and r_c can also be easily and reliably calculated without any instrument.

Based on the calculation examples and results of the synthetic, it can be concluded that the introduction of the new sample model into laboratory practice effectively facilitates the interpretation of measurement results and can be used for a better understanding of the behavior of rock samples originates from unconventional reservoirs.

It also makes it possible to determine the permeability (according to the applicability conditions) of rock samples that are damaged or too small to be measured in a standard permeameter.

The combination of traditional laboratory practice and new theses can increase the reliability of the measured petrophysical properties and significantly reduce the effect of damage due to storage casualties or sample preparation on the values.

The Crust Model Theory, which is a completely novel approach, can in many cases explain the productivity and production intensification issues and problems of domestic unconventional reservoirs.

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VIII. LIST OF PUBLICATIONS AND PRESENTATIONS

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